First Results from the Aberration-Corrected JEOL 2200FS-AC STEM/TEM

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The JEOL 2200FS electron microscope is the base platform on which JEOL's new generation of aberration-corrected STEM/TEM instruments is being constructed. The instrument operates at 200kV with a Schottky field emission electron source. It is designed to be operated solely via remote computer control, and so does not provide the standard viewing chamber with fluorescent screen, as no operator is required to sit adjacent to the column. The instrument incorporates an in-column energy filter of Omega geometry, and aberration correctors constructed by CEOS Co. (Heidelburg, Germany) in the condenser lens for STEM annular dark-field imaging and/or in the objective lens for TEM bright-field imaging. When an aberration corrector for the objective lens is fitted, a monochromator in the electron gun is available as an option [1], to allow control of the energy spread of the electron beam for more effective sub-Å imaging. Since the STEM image is not very sensitive to chromatic effects, a monochromator is typically not required for the STEM-corrected geometry, although it might be desired to allow higher energy resolution in energy-loss spectroscopy. Figure 1 shows the instrument configured with an aberration corrector included in the condenser; this is the geometry of instruments presently at ORNL and at Lehigh University.

Several different camera systems are provided to allow efficient computer operation from a separate room, in order to minimize operator presence on ultimate performance. All aperture drives, are operated by stepper motors, and aperture centering is done by observing the image from a Gatan 676 NTSC camera on a TV monitor. Wide-field digital images from a Hamamatsu 1M-pixel CCD camera positioned below the projector lens can be recorded for general TEM imaging, but high-resolution TEM images are better recorded on a Gatan 894 retractable 2048 x 2048px camera positioned below the camera chamber (film/imaging plates can still be used in the new instrument).

For TEM operation, depending upon gun operating parameters, a beam with energy spread from 1.3eV down to 0.7eV can be produced. Measured HT voltage stability of 0.6×10^{-6} (rms) and OL current stability of 0.25×10^{-6} (rms) give a defocus spread of 18.5 Å. At this value, the temporal envelope on the contrast transfer function allows information transfer to an information limit of about 0.85 Å (Fig. 2a). Sub-Å resolution can be accessed (in the absence of a TEM C_S-corrector) using the focal-series reconstruction (FSR) technique [2] at α -null [3] values of underfocus (Fig. 2b).

In STEM-corrected instruments, high-angle annular dark-field (or Z-contrast) imaging mode should allow a probe on the order of 0.7Å to be generated, yielding images with equivalent resolution. A caveat is that, because of the sensitivity of a scanning beam instrument to environmental influences that might affect the behavior of the probe, a laboratory room with highly controlled conditions must be provided. Magnetic fields less than 0.1mG are desired, along with room temperature variations within a 0.2°C/hr. Sample/stage stability are further enhanced for the side-entry specimen rod configuration by the provision of a "clamshell" chamber surrounding the specimen rod (see Fig. 1) that reduces the effects of pressure variations on image motion.

Native resolution in HA-ADF mode with no aberration correction, using the URP pole piece with Cs = 0.5mm and Cc = 1.1mm is 1.4Å. Figure 3a is a Z-contrast image of a Si single crystal in a <110> orientation, with characteristic "dumbbell" atomic column spacing of 1.36Å not fully resolved (image acquired during JEOL factory test-out, with environmental conditions significantly degraded from optimum). The corresponding Ronchigram, has an uncorrected flat phase field at 18mrad (delineated in Fig. 3b). After aberration correction, the corrected flat phase field extends to 35mrad (Fig. 3c). The improvement in resolution and contrast can be seen in the image of Fig. 3d. [5]

References

- [1] J.L. Hutchison et al., *JEOL News* **37E** (2002) 2-5.
- [2] W.M.J. Coene, A. Thust, M. Op de Beeck & D. Van Dyck, *Ultramicroscopy* **64** (1996) 109.
- [3] M.A. O'Keefe et al., *Ultramicroscopy* **89** (2001) 4: 215-241.
- [4] M.A. O'Keefe, Microscopy & Microanalysis 7, 2: (2001) 916.
- [5] Supported by Director, Office of Science, Office of Basic Energy Sciences, Materials Science Division, DOE (contract DE-AC03-76SF00098), and Asst. Sec. for EERE, Office of FreedomCAR and Vehicle Tech., HTML User Program, ORNL, managed by UT-Battelle, LLC for DOE (contract DE-AC05-00OR22725), and National Science Foundation, Division of Materials Sciences #9626279.

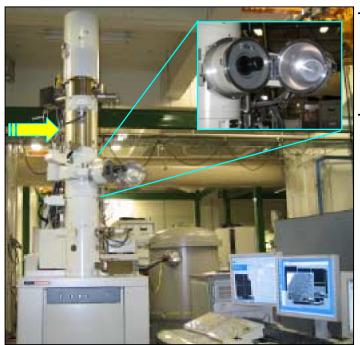


FIG. 1 JEOL 2200FS-AC with STEM corrector (arrow), and specimen rod "clamshell" inset.

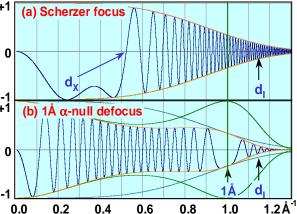


FIG. 2 Contrast transfer functions for TEM imaging mode of 2200FS-AC STEM/TEM.

- (a) Scherzer focus shows TEM resolution (d_X) to 1.9Å, with information transfer (d_1) to 0.86Å.
- (b) α -null defocus [4] maximizes transfer, in this case at 1Å, to enable sub-Ångstrom imaging in TEM mode using focal-series reconstruction.

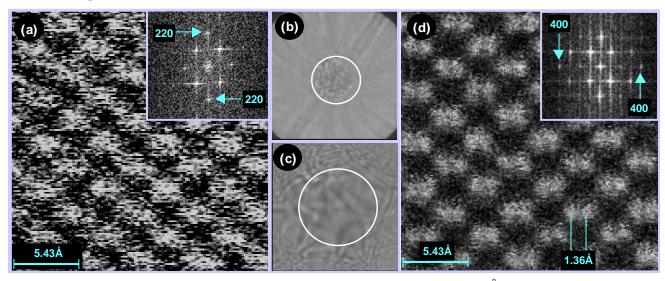


FIG. 3 (a) Z-contrast Si <110> image before C_s -correction (220 spacing at 1.92Å). Ronchigrams before (b) and after (c) probe correction. (d) Z-contrast Si <110> "dumbbell" image after correction (with 400 at 1.36Å).